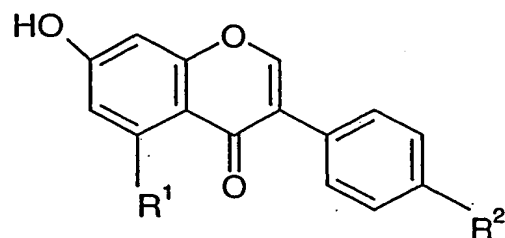


Claims

1. A process for manufacturing a hydroxylated isoflavone of the general formula

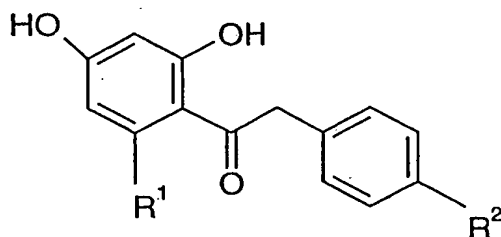


wherein R¹ signifies hydrogen or hydroxy, and

R² signifies hydroxy or C₁₋₆-alkoxy,

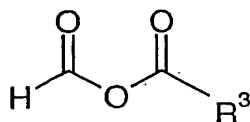
5

characterized by reacting a 2-hydroxydeoxybenzoin of the general formula



wherein R¹ and R² have the significances given above,

with a formic acid anhydride of the general formula



III

10 wherein R³ signifies C₂₋₂₀-alkyl, C₁₋₆-haloalkyl, (C₁₋₆-alkoxy)methyl, carboxy-C₂₋₆-alkyl, aryl-C₁₋₆-alkyl, a group -CH₂-(OCH₂CH₂)_m-O-C₁₋₆-alkyl, a group -CH(R⁴)=CR⁵R⁶, a group -CH=CH-COOH, C₃₋₈-cycloalkyl, aryl, heteroaryl, di(C₁₋₆-alkyl)aminomethyl, diarylaminomethyl, a group -(CH₂)_n-COOR⁷, a group -(CH₂)_m-COOCHO, a group -CH=CH-COOCHO, C₁₋₆-alkoxy, aryloxy or formyloxy,

15

each of R^4 , R^5 and R^6 , independently, signifies hydrogen, C_{1-6} -alkyl or aryl,

R^7 signifies hydrogen, C_{1-6} -alkyl or aryl,

m signifies an integer 1 to 4, and

n signifies zero or an integer 1 to 8,

5

in the presence of a base or in a solvent which acts as a base, and if necessary promoting the ensuing hydrolysis of the so-produced acylated form of the hydroxylated isoflavone of the formula I by acidification.

2. The process according to claim 1, wherein a base is employed and the process is
10 carried out additionally in an organic solvent.

3. The process according to claim 2, wherein the base is an alkali metal or alkaline
earth metal hydroxide, an alkali metal or alkaline earth metal carbonate or bicarbonate, an
alkali metal or alkaline earth metal salt of a carboxylic acid with up to 10 carbon atoms, an
aliphatic or mixed aliphatic/aromatic tertiary amine, a nitrogen-containing heterocyclic
15 base, or a secondary or tertiary phosphate.

4. The process according to claim 3, wherein the base is lithium, sodium or
potassium hydroxide, lithium, sodium or potassium carbonate, calcium or magnesium
carbonate, lithium, sodium or potassium bicarbonate, calcium or magnesium bicarbonate,
sodium formate, potassium propionate, trimethylamine, triethylamine, N-
20 ethyldiisopropylamine, N,N-dimethylethanolamine, 2-(dimethylamino)-ethyl acetate,
triethanolamine, N,N-dialkylaniline, optionally alkyl substituted pyridine, a N-alkyl
substituted piperidine, a N-alkyl substituted morpholine, imidazole, trisodium phosphate
or tripotassium phosphate.

5. The process according to claim 3 or 4, wherein the base is an alkali metal
25 carbonate, bicarbonate or formate, or an aliphatic or mixed aliphatic/aromatic tertiary
amine.

6. The process according to any one of claims 2 to 5, wherein the solvent is an
aliphatic or cyclic ether, a lower aliphatic nitrile, a lower aliphatic ester, dimethyl-
sulphoxide, a halogenated, particularly chlorinated, lower aliphatic hydrocarbon, an
30 aromatic hydrocarbon or a lower aliphatic ketone.

7. The process according to claim 6, wherein the solvent is diethyl ether,
diisopropyl ether, dibutyl ether, tert. butyl methyl ether, diethylene glycol dimethyl ether,
tetrahydrofuran, dioxan, acetonitrile, a lower alkyl formate or acetate, dimethylsulphoxide,

methylen chloride, benzene, toluene, an xylene, acetone, 2-butanone, diethyl ketone or methyl isobutyl ketone.

8. The process according to claim 1, wherein the process is carried out in a solvent which acts as a base, and said solvent is a di(lower alkyl)formamide, preferably
5 dimethylformamide, dimethylacetamide, tetramethylurea or N-methyl-pyrrolidone.

9. The process according to any one of claims 1 to 8, wherein the process is carried out at temperatures in the range of about -20°C to about +80°C, preferably at temperatures from about -5°C to about +45°C.

10. The process according to any one of claims 1 to 9, wherein about 1.5 to about
10 6 equivalents, preferably about 2.5 to about 5 equivalents, of the formic acid anhydride of the formula III are present in the reaction mixture per equivalent of the 2-hydroxydeoxybenzoin of the formula II.

11. The process according to any one of claims 2 to 7, 9 and 10, wherein the base is present in the reaction mixture in an amount which is up to about 6 equivalents per
15 equivalent of the 2-hydroxydeoxybenzoin of the formula II.

12. The process according to any one of claims 1 to 11, wherein the hydrolysis of any produced acylated form of the desired hydroxylated isoflavone of the formula I in the mixture after reaction is effected by addition of aqueous mineral acid to lower the pH of the mixture to about 0-2, preferably about 0-1, and heating the acidified mixture.

13. The process according to claim 12, wherein the solvent is continually removed
20 by distillation during the hydrolysis and continually replaced with a lower alkanol, preferably methanol or ethanol, or with water to dilute the mixture, or wherein as much as possible of the solvent is first distilled off, optionally under reduced pressure, and the lower alkanol or water added thereafter, and in either case further acid is then optionally
25 added to the mixture to restore the pH-value to about 1-2, preferably about 0-1, and the mixture is heated, preferably at reflux temperature, for a further period to complete the hydrolysis.

14. The process according to any one of claims 1 to 13, wherein the desired product of the formula I is isolated, either directly from the mixture on completion of the
30 reaction of the formic acid anhydride of the formula III with the 2-hydroxydeoxybenzoin of the formula II or following the subsequent acid-catalysed hydrolysis for deacylation, by crystallization induced by addition of water and removal of the resulting crystalline product by filtration.

15. The process according to any one of claims 1 to 14, wherein propionic formic anhydride is used as the formic acid anhydride of the formula III.

16. The process according to any one of claims 1 to 14, wherein isobutyric formic anhydride, carbonic monoformic anhydride methyl ester or carbonic monoformic
5 anhydride ethyl ester is used as the formic acid anhydride of the formula III.
